

TRANSLATION INTO ENGLISH OF:

SUBSTANTIATION OF THE OPPOSITION FILED ON
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OPPOSITION AGAINST

**European patent no. 0 803 593 (application no. 97 106 815.0)
in full scope by virtue of Article 100a), 54 and 56 EPC**

Title: "Method of preparing group III-V compound semiconductor crystal"

Proprietor: SUMITOMO Electric Industries, Ltd., Osaka-shi, Osaka 541 JP

Opponent: Freiburger Compound Materials GmbH, Am Junger Löwe Schacht 5, D-09599 Freiberg, Germany

SUBSTANTIATION

I. Ground for opposition: lack of novelty, Article 100a and Article 54(2), EPC

1. Claim 1

Claim 1 of the patent in dispute refers to a method comprising the following features:

- (a) A group III-V compound semiconductor crystal containing a doped element is prepared.
- (b) A crucible or boat (12) is filled with
 - (b1) a pre-synthesized III-V compound raw material (2),
 - (b2) solid carbon (13, 23, 43), and
 - (b3) boron oxide (4).
- (c) The crucible or boat (1) filled with the compound raw material (2), the solid carbon (13, 23, 43), and the boron oxide (4) is sealed within an airtight vessel (5) formed of a gas impermeable material.
- (d) The compound material (2) is heated and melted in the sealed state within the airtight vessel (5).
- (e) The melted material (2) is solidified to grow a carbon-doped compound semiconductor crystal.

2. Lack of novelty in the light of D1 (cf. Frank and K. Hein, Cryst. Res. Technol. 30 (1995), pages 897-909)

As will be shown hereinafter, D1 discloses all features (a) through (e) of claim 1. It should be noted that the content of D1A (Hein et al., Metall 47 (1993), pages 924-928), specifically sections 3.1 and 3.2 which refer to the vertical gradient freezing (VGF) method, is fully comprised by the disclosure of D1, since D1 directly refers to D1A in connection with the use of such a VGF method, which in D1 is described for the growth experiments thereof (page 898, second paragraph, "Experimental" of D1). Accordingly D1 discloses the following features:

Feature (a):

A GaAs- (III-V compound semiconductor) crystal is prepared (title and summary).

Feature (b):

A crucible of pBN is filled with pre-synthesized GaAs (D1, page 989 "Experimental"). Solid carbon (ultrapure graphite disk) was introduced into the growth system (VGF crystal no. 51 and 52 in Tables 6 and 9; page 905, lines 1-2 of the text; page 907, lines 1-2), i.e., it was filled into the crucible for additional carbon stabilization and for setting the carbon doping concentration. Furthermore, boron oxide (B_2O_3) has been filled into the crucible for obtaining a B_2O_3 melt (section 3.5 "Results using B_2O_3 encapsulation" on pages 905-907; VGF crystal no. 52 in Table 9; last paragraph on page 909). That is why features (b1), (b2) and (b3) are disclosed in D1.

Features (c) through (e):

It follows from document D1A (see sections 3.1 and 3.2, as well as Figs. 2 and 3 on pages 925-927, particularly page 925, right column and Fig. 3) that the VGF-prepared GaAs crystals of D1 were prepared in the following steps:

- the crucible which is filled with the compound raw material, solid carbon and boron oxide is sealed in an airtight vessel formed of a gas impermeable material (quartz ampoule),
- the compound material is heated and melted in the sealed state within the airtight vessel, and
- the melted compound material is solidified to grow a carbon-doped compound semiconductor crystal.

That is why all features (a) to (e) of claim 1 are disclosed by D1/D1A, and the subject matter of claim 1 is thus not novel.

3. Lack of novelty with respect to D2 (FE project TK 0378/5)

3.1 Document D2 is a publication that has been available to the public under signature F 95 B 2529 in the library of the University of Hanover since February 19, 1996. The enclosed letter from the library of the University of Hanover (D2A) confirms the publishing date of February 19, 1996. That is why D2 is prior art under Article 54(2) EPC.

3.2 Document D2 discloses the following:

Feature (a):

A method of preparing a GaAs-(III-V compound) semiconductor having carbon doped (page 64, section 6.2.4.2 "Solid Doping with Carbon").

Feature (b):

A crucible of pBN (section 6, "Single-crystal Growth", on pages 40-48, particularly page 42, first paragraph, and page 46, penultimate paragraph) is filled with pre-synthesized GaAs, solid carbon and boron oxide (Title: "Solid doping with Carbon", and lines 1-8 of page 64, first paragraph of page 65; sample E1 in Table 6.2.4.2.1) whereby features (b1), (b2), and (b3) are anticipated.

Features (c) through (e):

Further details regarding the preparation of the GaAs crystal doped with solid carbon are described in the description of the crystal growth process on pages 40-48. Hence, it follows that

- the crucible filled with the compound raw material, the solid carbon and the boron oxide is sealed within an airtight vessel formed of a gas impermeable material (this becomes inter alia apparent from the fact that an inert gas atmosphere is produced; see page 40, line 16; middle and the last three lines of page 43; page 45, lines 1-3 and last paragraph),

- the compound material is heated and melted in the sealed state within the airtight vessel (see the descriptions regarding the melted system in the furnace or heater; e.g. page 40, last paragraph; page 42, third paragraph; middle of page 43; page 46, last paragraph), and
- the melted compound material is solidified to grow a carbon-doped compound semiconductor (GaAs) crystal (page 46, last paragraph; page 65 (Table 6.2.4.2.1)).

Hence, all features (a) through (e) are disclosed in D2, and the subject matter of claim 1 is thus also not novel in the light of D2.

4. Sub-claims 2 to 14

4.1 Claim 2

When the solid carbon is filled together with the GaAs material and the boron oxide into the crucible, the heat-melted boron oxide is necessarily brought into contact with at least part of the solid plastics in the heating/melting step. This is e.g. confirmed by the statement in D1 (legend of Table 9 on page 906) that use is made of a B_2O_3 melt and a carbon source for a certain weight amount of 250 g GaAs, and that the carbon concentration C_{As} in the VGF-produced crystal is influenced by the carbon source incorporated into the growth system (page 907, lines 1-2).

Furthermore, it should be noted that the reactions described on page 66 of D2, of which it is assumed that they occur when solid carbon is added to GaAs in the presence of boron oxide, are only possible if the heat-melted boron oxide is brought into contact with at least part of the solid carbon during the heating/melting step.

Moreover, the applicant himself points out in the response of March 9, 2001, paragraph 6.1, on page 4 that the contact between residual water containing boron oxide and solid carbon is automatically established by incorporating the pre-synthesized III-V compound raw material, the boron oxide and the solid carbon into a sealed crucible and by heating the same. Since D1 and D2 use the same system, such a contact is also bound to be established automatically.

Claim 2 is thus not novel in the light of D1 and D2.

4.2 Claims 3 and 4

It is generally known that B_2O_3 , which is used in D1 and D2 as a boron oxide material, contains residual water. This is e.g. evidenced by D2 on page 35 which discusses the influence of the water content that is contained in the boron oxide used. D2 actually points out on page 65 that the water contained in the boron oxide may have an influence as to how the added solid carbon in GaAs is doped (this involves the oxidation of carbon by water to CO for doping GaAs). Hence, the feature is anticipated by each of the disclosures in D1 and D2.

Since it must be assumed that a water content of 10-500 wt ppm corresponds to a typical residual content, claim 4 is not novel either.

4.3 Claim 5

Since a graphite disk was used in D1 as a carbon source, i.e. a material which is handled as bulk material, the amount of the filled-in solid carbon in D1 is bound to be greater than the amount of carbon doped into the GaAs crystal, which is in the order of $10^{14} - 10^{15} \text{ at/cm}^3$ (see Tables 6-9).

D2 (see the paragraph on page 64, particularly lines 1-4 from below) provides for amounts of solid carbon in the order of 10 µg to obtain a doping concentration of 1×10^{15} at/cm³, which corresponds to 0.000004 g = 4 µg C per 1 kg GaAs. Furthermore, it is pointed out that in general the measured C concentration in the crystal was clearly below the value to be expected from the addition.

Claim 5 is thus not novel in the light of D1 or D2.

4.4 Claim 6

The expert reader of D1 who is instructed to use an ultrapure graphite disk as the carbon source (page 905, lines 1-2) will note that said solid carbon has been subjected to a heat treatment under reduced pressure to achieve the purpose described on page 4, line 5 of the patent in dispute. That is why the feature of claim 6 is anticipated by D1.

4.5 Claims 7 and 8

It is self-evident that the melted GaAs material is kept in the melted state for a specific period of time before being solidified. Hence, the feature of claim 7 is automatically fulfilled in the systems of D1 and D2. This becomes e.g. apparent from the expression "GaAs melt", which is repeatedly used in D1 and D2. Furthermore, it follows from the description of the growth conditions for the GaAs crystal in section 3.2 of D1A, the conditions having been applied in the production method of D1 (see above all the growth rate values described in the right column of page 926 and in Fig. 5 of D1A), that the molten GaAs is kept in the melted state for several hours.

Therefore, the subject matters of claims 7 and 8 are not novel.

4.6 Claim 9

The description of D2 (page 64) on the use of solid carbon amounts in the order of 10 µg is clearly understood by the expert reader such that carbon is added in powder form.

Claim 9 is thus not novel in the light of D2.

4.7 Claims 11 and 12

The use of an ultrapure graphite disk as a carbon source in D1 anticipates the feature that the solid carbon comprises bulk carbon, specifically a sintered compact of carbon powder, because a graphite disk is obtained by sintering graphite powder.

Hence, the subject matters of claims 11 and 12 are not novel in the light of D1.

4.8 Claims 13 and 14

As shown by the above discussion under items 2 and 3, both D1 and D2 describe the use of a pBN crucible and the preparation of a GaAs crystal as the III-V compound semiconductor crystal. Therefore, claims 13 and 14 are not novel either.

II. Ground for opposition: lack of an inventive step, Article 100(a) and Article 56 EPC

1. As shown above under items I.2 and I.3, both D1 and D2 disclose the central feature of the patent in dispute, i.e. to add solid carbon and boron oxide to a pre-synthesized GaAs (III-V compound) raw material to prepare a GaAs crystal with a well-set doped carbon concentration. The other features of claim 1 and those of claims 2 to 9 and 11 to 14 are also disclosed in D1/D1A and D2, respectively. That is why an amendment to claim 1 for establishing novelty in a formal way would lead to a claimed subject matter that would be rendered obvious either by D1 or D2 and would therefore lack an inventive step. For instance, the use of carbon fiber according to claim 10 instead of the graphite disk used in D1 or the carbon powder used in D2 would just offer an alternative to one skilled in the art. Such an alternative is obvious and cannot support any inventive step.

2. Lack of an inventive step on the basis of a combination of D3 (US 4,999,082) with D1 or D2

2.1 Document D3, Example 2 (column 8, line 45 through column 9, line 7; Fig. 2), discloses a method of preparing a GaAs (III-V compound) semiconductor crystal having carbon doped, the method comprising the following steps:

- filling the crucible with pre-synthesized GaAs raw material,
- sealing the crucible filled with the GaAs together with a carbon disk placed on the opening of the crucible, within an airtight vessel formed of a gas impermeable material (sealed quartz ampoule),
- heating and melting the GaAs material in the sealed state within the airtight vessel, and
- solidifying the melted GaAs material to grow the carbon-doped GaAs crystal.

D3 further discloses in Example 2 (column 8, lines 53-56) that the amount of carbon incorporated into LEC-produced GaAs may be controlled by varying the water content of the B_2O_3 encapsulant used in the process.

Therefore, the only difference between D3 and claim 1 of the patent in dispute is that the solid carbon is not directly filled together with the boron oxide into the crucible. Nevertheless, the solid carbon is introduced within the airtight vessel (sealed quartz ampoule) to be in fluid communication with the pre-synthesized GaAs compound (cf. column 5, lines 43-45).

- 2.2 The objective object underlying the patent in dispute, as compared with D3, can be seen in the provision of a III-V compound (GaAs) semiconductor crystal in the case of which the amount of doped carbon can easily be set during crystal growth (see paragraph [0027] of the patent in dispute).
- 2.3 As stated above under item 1.2, document D1 discloses that the addition of solid carbon into the growth system together with boron oxide, i.e. within the crucible containing the pre-synthesized GaAs raw material, leads to stabilized carbon doping and an easily adjustable carbon doping concentration and thus to a good control of the desired specific electrical resistance in dependence upon the carbon concentration (see especially section 3.5 on pages 905 to 908).

Therefore, D1 clearly points out that solid carbon should be added together with boron oxide directly to the pre-synthesized GaAs contained in the crucible, instead of just being in fluid communication with GaAs, for achieving an advantageous setting of the carbon doping during crystal growth, and thus for solving the problem mentioned under item 2.2 above. Claim 1 is thus rendered obvious by a combination of documents D3 and D1.

- 2.4 Document D2 points just as clearly to an easy setting and control of the C doping process and thus of the specific electrical resistance by directly doping solid C to the pre-synthesized GaAs in the presence of boron oxide (section 6.2.4 on pages 62 to 63 and section 6.2.4.2 on pages 65 and 66).

Therefore, the subject matter of claim 1 is also rendered obvious and is devoid of an inventive step when documents D3 and D2 are combined.

- 2.5 The features of the dependent claims 2 to 14 are also known or within the scope of routine skill of a person skilled in the art in the light of the disclosures of D1, D2 and/or D3 and can thus not support an inventive step either.

III. Summary

In the light of the above assessment the patent in dispute must be revoked in full scope on the basis of the substantiated ground for opposition of Article 100 (a) EPC because of lack of novelty and lack of an inventive step.

[Signature]